

Supplementary Information:

Chemistry of Spontaneous Alkylation of Methimazole with 1,2-Dichloroethane

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Table S1. Crystal data and structure refinement for **4a**, **4b**, **6** and **8**.

	4a	4b	6	8
Molecular formula	[C ₆ H ₉ N ₂ S]Cl	[C ₆ H ₉ N ₂ S]BF ₄	[(C ₆ H ₉ ClN ₂ S) ₂ Ag]BF ₄	C ₁₀ H ₁₄ N ₄ S ₂
Formula weight	176.66	228.02	548.00	254.37
Temperature/K	293(2)	169.99(10)	169.99(10)	169.99(10)
Crystal system	Orthorhombic	Monoclinic	Monoclinic	Monoclinic
Space group	<i>Pnma</i>	<i>P2₁/c</i>	<i>P2₁/c</i>	<i>P2₁/c</i>
<i>a</i> /Å	12.6091(7)	5.56720(10)	7.54590(10)	12.1442(5)
<i>b</i> /Å	6.5090(5)	17.1985(4)	13.5083(2)	6.9640(3)
<i>c</i> /Å	9.9212(5)	10.0040(2)	19.3375(3)	14.1111(6)
α /°	90	90	90	90
β /°	90	93.848(2)	93.3310(10)	93.260(4)
γ /°	90	90	90	90
Volume/Å ³	814.26(9)	955.70(3)	1967.79(5)	1191.48(9)
<i>Z</i>	4	4	4	4
ρ_{calc} /g cm ⁻³	1.441	1.585	1.850	1.418
μ /mm ⁻¹	0.650	3.304	13.098	3.874
<i>F</i> (000)	368.0	464.0	1088.0	536.0
Radiation	Mo K α (λ = 0.71073)	Cu K α (λ = 1.54184 Å)	Cu K α (λ = 1.54184)	Cu K α (λ = 1.54184)
2 θ range for data collection/°	8.156 to 52.998	10.246 to 132.79	7.988 to 132.996	7.292 to 133.898
Index ranges	-15 ≤ <i>h</i> ≤ 15, -5 ≤ <i>k</i> ≤ 8, -11 ≤ <i>l</i> ≤ 12	-6 ≤ <i>h</i> ≤ 5, -20 ≤ <i>k</i> ≤ 20, -11 ≤ <i>l</i> ≤ 11	-8 ≤ <i>h</i> ≤ 8, -16 ≤ <i>k</i> ≤ 16, -22 ≤ <i>l</i> ≤ 20	-14 ≤ <i>h</i> ≤ 14, -5 ≤ <i>k</i> ≤ 8, -16 ≤ <i>l</i> ≤ 16
Reflections collected	3050	10548	15351	8232
Independent reflections	914 [<i>R</i> _{int} = 0.0371, <i>R</i> _{sigma} = 0.0350]	1689 [<i>R</i> _{int} = 0.0595, <i>R</i> _{sigma} = 0.0374]	3450 [<i>R</i> _{int} = 0.0343, <i>R</i> _{sigma} = 0.0281]	2117 [<i>R</i> _{int} = 0.0305, <i>R</i> _{sigma} = 0.0260]
Data/restraints/parameters	914/0/62	1689/256/174	3450/0/237	2117/0/148

Goodness-of-fit on F^2	1.083	1.064	1.084	1.076
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0333$, $wR_2 = 0.0824$	$R_1 = 0.0777$, $wR_2 = 0.1856$	$R_1 = 0.0247$, $wR_2 = 0.0641$	$R_1 = 0.0298$, $wR_2 = 0.0803$
Final R indexes [all data]	$R_1 = 0.0391$, $wR_2 = 0.0868$	$R_1 = 0.0798$, $wR_2 = 0.1872$	$R_1 = 0.0262$, $wR_2 = 0.0649$	$R_1 = 0.0321$, $wR_2 = 0.0823$
Largest diff. peak/hole / e \AA^{-3}	0.38/−0.32	0.53/−0.42	0.56/−0.71	0.23/−0.25
CCDC	2105605	2105606	2105607	2105608